Torrefaction of Rubberwood Waste: The Effects of Particle Size, Temperature & Residence Time

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Highlights:

- Rubberwood waste in shredded and woodchip form was torrefied.
- The torrefaction process of rubberwood waste is affected by particle size, temperature and residence time.
- Torrefaction of woodchip rubberwood at 300 °C with a residence time of 60 minutes was the optimum condition.
- The properties of the optimum biochar product were close to those of subbituminous rank coal.

Abstract. Agriculture waste has created massive challenges over the last few decades and yet also opportunities. This work aimed to produce high-quality biochar from rubberwood waste with calorific properties close to subbituminous coal. Using a tubular vertical reactor, the effects of rubberwood particle size (wood chips and shredded wood), torrefaction temperature (220, 260, and 300 °C), and residence time (30, 60, and 90 minutes) on the quality of torrefied rubberwood were studied. The results showed that the mass loss of the rubberwood increased as the temperature increased. Also, the particle size and residence time increased due to excessive devolatilization. A higher fixed-carbon content and calorific value as well as lower moisture and volatile-matter content in the biochar were achieved by increasing the torrefaction temperature and residence time in comparison to the untreated sample (raw rubberwood). The highest fixed-carbon content and calorific value were found to be 56.7% and 6313 kcal/kg, respectively, for the wood chip particles that were torrefied at 300 °C for 60 minutes. Based on the Van Krevelen diagram, torrefaction of woodchip rubberwood at 300 °C with a residence time of 60 minutes demonstrated the optimum condition to generate a product with properties that are close to those of subbituminous rank coal.

Keywords: *calorific value; particle size; residence time; rubberwood waste; temperature; torrefaction.*

1 Introduction

With the fourth highest population number in the world, Indonesia contributes 4.47% of total world greenhouse gas emissions, attributed to deforestation,

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peatland degradation, forest fires, and in particular industrial growth, which consumes large amounts of fossil fuels as the power source [1]. Indonesia also produces large amounts of biomass wastes, equivalent to 470 GJ per year [2]. A lot of attention is drawn to the use of biomass as a renewable energy source. However, using biomass in its natural state has not proven to be an efficient technique due to its low density, high moisture content and hydrophilic nature, which has given rise to a wide range of processing treatments to improve the properties of biomass as an energy source [3]. Therefore, the utilization of biomass waste using an appropriate technology is expected to be able to contribute to solving environmental and energy problems.

Biomass is generally defined as renewable organic matter that is derived from living organisms [4]. From a renewable energy perspective, the term 'biomass' refers to materials that are commonly harvested from plants [5]. Biomass contains lignocellulosic and non-lignocellulosic materials. Lignocellulosic materials consist of cellulose, hemicellulose, and lignin [4], while non-lignocellulosic materials consist of non-cellulosic organic matter and are mainly used for nutritional purposes [6]. Biomass is typically acclaimed as a 'carbon neutral' fuel since biomass is part of the bio-cycle. Therefore, biomass can be also referred to as a sustainable material that can generate much lower net carbon emissions compared to fossil fuel [7].

Rubberwood waste is considered as a potential biomass feedstock in Indonesia. Based on the data from the Central Bureau of Statistics, Indonesia's rubber plantation area was 3.64 million hectares in 2016 [8]. With an economic lifetime of around 25-30 years, rubber trees are cut down each year for replantation, leading to an average annual production of 14.7-19.6 million tons of rubberwood [9]. Data from the Directorate General of Plantations in 2012 shows that 3% of the rubberwood plantations in Indonesia are replanted annually, resulting in approximately 844,500 m^3 of rubberwood waste. Currently, a large part of the rubberwood waste is underutilized, with a small fraction used as a power source in households or home industries and as a material for building furniture; thus it is considered to be a potential biomass-based energy source [10].

While biomass can be used as an alternative power source, direct combustion of biomass is not adopted by major industries [11]. Owing to the large difference between biomass and coal combustion properties, such as grindability and biodeterioration, it is difficult to replace fossil fuel with biomass without any major performance consequences [4]. One of the processes that could be used to increase the viability and combustion properties of biomass is torrefaction. Torrefaction is a thermolysis process of biomass in the absence oxygen at a low heating rate (< 50 °C/min) and a relatively low temperature range of 200 to 300 ºC [4,12]. The torrefaction treatment process comprises moisture vaporization,

devolatilization and depolymerization reactions to the biomass [6]. It is based on the removal of oxygen from the biomass, aiming to generate a fuel with higher energy density by decomposing the reactive hemicellulose content. Torrefaction also releases condensable matter in the form of tar, oil and water, and various gases including carbon monoxide, carbon dioxide and hydrogen.

Considering the importance of biomass waste utilization and torrefaction, this work aimed to examine the prospect of rubberwood utilization as biomass waste to be further upgraded to biofuel by torrefaction. Several important parameters, i.e. rubberwood particle size and torrefaction process conditions (residence time and torrefaction temperature) were investigated.

2 Methodology

2.1 Materials and Apparatus

The sample used in this work was rubberwood waste originating from Indonesia in the form of shredded wood and wood chips. The size of the rubberwood chips and the shredded rubberwood was in the range of 0.1 to 2 cm^3. A schematic of the torrefaction apparatus is shown in Figure 1. It consists of a vertical tubular reactor, a temperature controller, a thermocouple, a desiccator, plastic tubing, a nitrogen tank, and a rotameter. An electronic scale was used for weighing the samples before and after treatment.

2.2 Characterization

Proximate analysis (fixed carbon, moisture, volatile matter, and ash) and ultimate analysis (carbon (C), oxygen (O), hydrogen (H), nitrogen (N), and sulfur (S)) was conducted according to the ASTM D3172 and ASTM D3176 standard practice, respectively, at the Centre for Research and Development for Mineral and Coal Technology.

In brief, proximate analysis is done in three stages of temperature increase: (i) the sample is put inside a platinum crucible, which is placed inside a furnace. In order to remove the moisture from the sample, the starting temperature of 30 °C is increased to 110 °C at a rate of 30 °C per min. (ii) The temperature is increased to 900 °C at a rate of 30 °C per min in a nitrogen-rich environment (to prevent combustion). It is assumed that after this period all volatile components have been released. (iii) The sample is maintained at 900 °C in a now oxygen-rich environment for one minute so that the sample can be completely combusted. The remaining weight in the crucible is considered the ash content. The difference in weight between the end of stage (ii) and (iii) is considered the fixed carbon content.

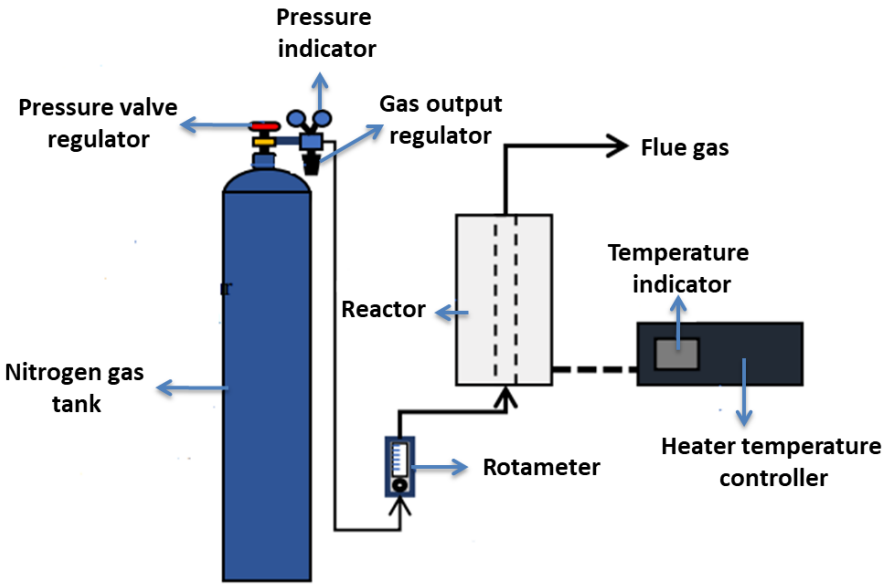

Figure 1 Schematic of torrefaction apparatus**.**

2.3 Torrefaction of Rubberwood

Torrefaction was conducted in a vertical tubular furnace under inert conditions and atmospheric pressure. Initially, the rubberwood samples were fed into the furnace. The furnace was then heated up until it reached the torrefaction temperature (varied at 220 °C, 260 °C, and 300 °C). This temperature range was chosen to represent mild pyrolysis [13]. The temperature of the furnace was measured by a thermocouple and controlled to the desired temperature by a controller. After the torrefaction process was completed within a certain residence time (varied at 30, 60, and 90 minutes with a torrefaction temperature of 260 °C and at 60 minutes with a torrefaction temperature of 220 °C and 300 °C), the furnace was cooled down. The torrefied rubberwood product was collected and placed in a desiccator until it reached room temperature and then weighed. The product characteristics were evaluated by calorific value, proximate and ultimate analysis, and Hardgrove Grindability Index (HGI) analysis.

3 Results and Discussion

3.1 Physical Appearance of Torrefied Rubberwood

The physical appearance of torrefied rubberwood is characterized by a color change compared to raw rubberwood (Figure 2). The color change was obtained

as a function of temperature, residence time, and particle size of the rubberwood. The color of the shredded rubberwood and the woodchip rubberwood used as raw materials is shown in Figure 2(a) and Figure 2(b), respectively, while the color of the torrefied shredded rubberwood and the torrefied woodchip rubberwood is shown in Figure 2(c) and Figure 2(d), respectively. It can be seen that the color of the raw rubberwood was brown.

With an increase in temperature and residence time, it can be seen that the torrefied rubberwood turned darker from raw brown to dark brown at a torrefaction temperature of 220 °C with a residence time of 60 minutes and from dark brown to dark (black) at a torrefaction temperature above 260 °C with a residence time of 60 minutes. The color changes of the torrefied rubberwood have been reported to be the result of hemicellulose degradation in the temperature range of 200 to 270 °C [14]. Within that range, the torrefaction reaction becomes more exothermic, hence releasing moisture content, carbon dioxide, and a large amount of acetic acid with some phenols that have low energy values [15].

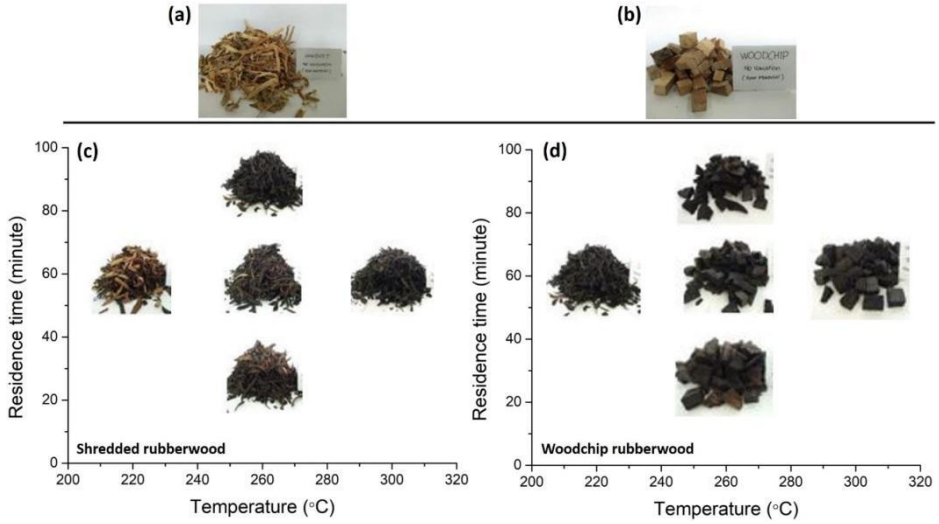

Figure 2 Physical appearances of raw rubberwood: (a) shredded rubberwood, (b) woodchip rubberwood, and torrefied (c) shredded rubberwood, (d) woodchip rubberwood, as a function of temperature and residence time.

3.2 Mass Loss and Gross Calorific Value Analysis

During torrefaction, rubberwood loses its mass as a function of the increase in temperature and residence time, as shown in Figure 3. In this case, temperature is as significant as residence time in determining the mass loss. The results in

Figure 3 show that for the lowest temperature of 220 °C, the mass loss was around 20% for the shredded rubberwood and the woodchip rubberwood.

As the temperature increased to 260 °C and 300 °C, the mass loss increased by approximately 1.5 and 2.5 times for the shredded rubberwood and around 1.5 and 3 times for the woodchip rubberwood, respectively.

In the treatment at 260 °C, there was a slight enhancement of mass loss from 30 to 40% by increasing the residence time from 30 to 90 minutes for the woodchip rubberwood, while there was a significant mass loss increment (around 3-fold) for the shredded rubberwood at a residence time of 90 minutes compared to a residence time of 20 minutes. This may be due to the missing volatile matter having a high C/H ratio at extended reaction time. This assumption is supported by the ultimate analysis, which was not significantly different, particularly in elemental carbon content and hence the GCV looks similar, with a value of 4880 and 4766 kcal/kg for residence time at 60 and 90 minutes, respectively (Figure 4).

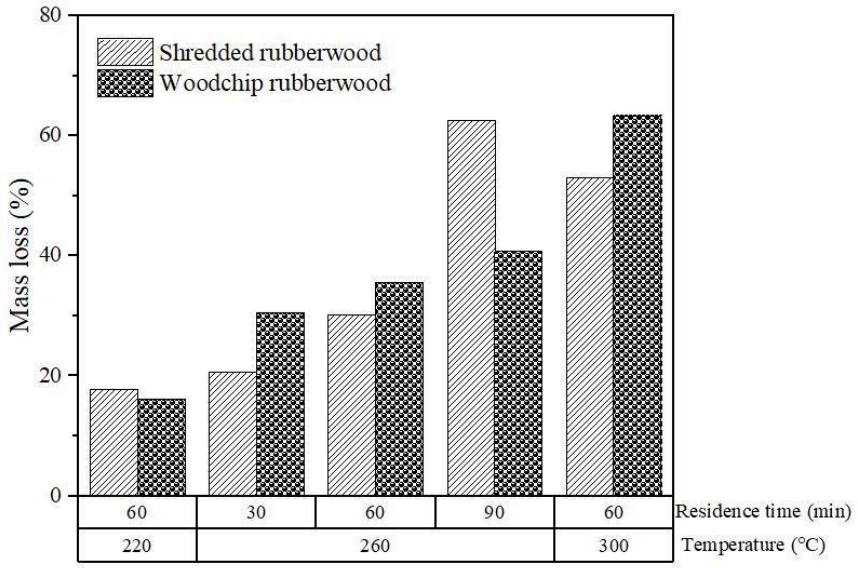

Figure 3 Mass loss of the torrefied shredded rubberwood and woodchip rubberwood as a function of temperature and residence time.

The trend of mass loss of the shredded rubberwood was almost the same as in the work reported by Felfli, *et al.* [16], who used wood briquettes as biomass source and showed that a significant increment (almost 3-fold) was observed when the wood briquettes were torrefied at a torrefaction temperature of 220 °C with a

residence time of 90 minutes compared to a residence time of 60 minutes. It was reported that the mass loss was triggered by hemicellulose, lignin and cellulose degradation at 150, 250 and 275°C, respectively [4]. Consequently, the volatilization of volatile matter and inherent moisture increased as the torrefaction temperature and residence time were increased.

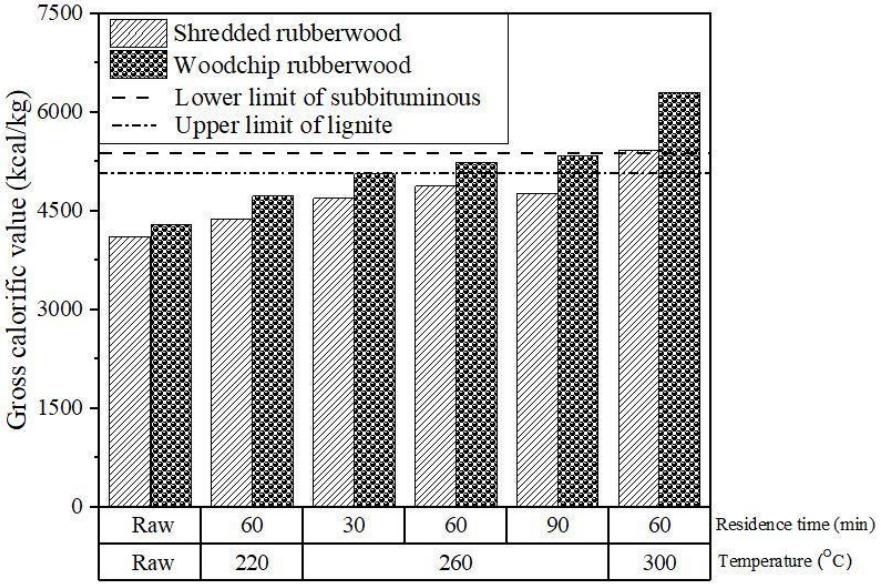

Figure 4 Gross calorific value of the torrefied shredded and woodchip rubberwood compared to the raw shredded rubberwood and woodchip rubberwood, lignite, and subbituminous coal.

Gross calorific value (GCV) is one of the most important parameters for predicting the behavior of biochar/torrefied rubberwood, defining rank, evaluating its potential use in the combustion system, and evaluating pollution compliance. Figure 4 shows that the calorific value was directly proportional to the torrefaction temperature and residence time, respectively. The GCV was observed to consistently increase with increasing torrefaction temperature and residence time. The GCV of the shredded and woodchip rubberwood at a temperature of 220 °C with a residence time of 60 minutes was 4377 and 4728 kcal/kg (6% and 10% higher compared to untorrefied rubberwood), respectively.

By increasing the temperature to 260 and 300 °C, the GCV of the shredded rubberwood increased by 11% and 24%, respectively, while for the woodchip rubberwood, there was an increase of GCV by 11% and 33% compared to its raw material, respectively. For the torrefaction at 260 °C, there was a slightly increased GCV, from 5068 to 5351 kcal/kg, at longer residence time, from 30 to

90 minutes, for the woodchip rubberwood. Meanwhile, for the shredded rubberwood there was a fluctuating trend of GCV that maximized at a residence time of 60 minutes with GCV at 4880 kcal/kg.

The result shows that the highest GCV of torrefied rubberwood was achieved for the woodchip rubberwood that was torrefied at a temperature of 300 °C with a residence time of 60 minutes, with GCV at 6313 kcal/kg. This product can be categorized as subbituminous rank coal. The GCV trend of the woodchip rubberwood in this work was almost the same as in the work reported by Felfli, *et al.* [16], who used wood briquettes as biomass source.

At low-temperature torrefaction, i.e. < 250 °C, the main decomposition of rubberwood comes from limited devolatilization and carbonization of hemicellulose [17]. Minor decomposition is expected for lignin and cellulose, except for chemical changes in their structure, resulting in less significant mass loss. At higher torrefaction temperature and residence time, i.e. > 250 °C and > 30 minutes, decomposition of rubberwood becomes more vigorous as hemicellulose extensively decomposes into volatiles and a char-like solid product while lignin and cellulose show limited devolatilization and carbonization [18]. During the torrefaction process, rubberwood undergoes volatilization of hydrogen and oxygen atoms rather than carbon atoms, resulting lower H/C and O/C ratios [14]. In that condition, torrefied rubberwood is more hydrophobic with higher energy density with a calorific value similar to that of lignite coal.

3.3 Proximate Analysis

The moisture content, volatile matter, ash, and fixed carbon of shredded and woodchip rubberwood can be obtained by proximate analysis, as shown in Figure 5. It is apparent that the torrefied rubberwood had higher fixed carbon content but lower volatile matter and moisture content due to devolatilization compared to the raw rubberwood. On the other hand, the amount of ash remained constant after torrefaction.

The torrefied shredded rubberwood and woodchip rubberwood had higher fixed carbon content with a 1.1- and 1.4-fold increment compared to the raw rubberwood (Figure 5(a)). The enhancement of fixed carbon content in the torrefied rubberwood was affected by carbonization of hemicellulose at 200 to 250 °C along with lignin and cellulose at 250 to 300 °C. This can also be attributed to the loss of volatile matter from the rubberwood. With the decrease of the overall rubberwood mass, the amount of fixed carbon content increased, and with increasing residence time, more carbonization and devolatilization occurred. Interestingly, the fixed carbon content increased sharply at a

torrefaction temperature of 300 °C, which is caused by devolatilization of both cellulose and lignin along with more and faster carbonization [14].

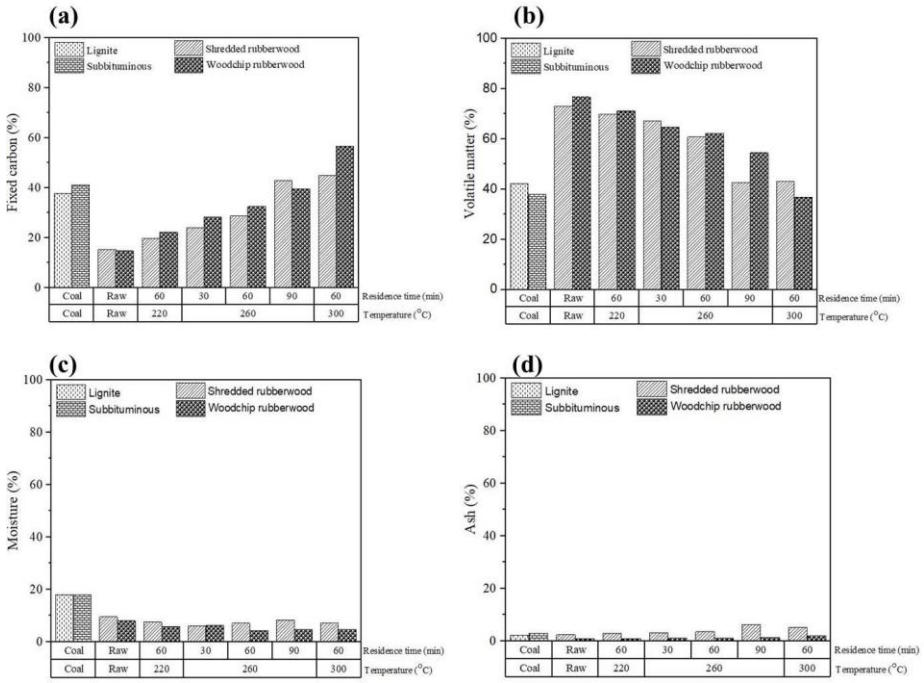

Figure 5 Analysis of (a) fixed carbon, (b) volatile matter, (c) moisture, (d) ash of the torrefied shredded rubberwood and the woodchip rubberwood compared to the raw shredded rubberwood and woodchip rubberwood, lignite, and subbituminous coal.

The torrefied shredded rubberwood and woodchip rubberwood had lower volatile matter content compared to the raw rubberwood with a reduction of 22.46% and 24.60%, respectively (Figure 5(b)). In this case, with an increase of residence time, the amount of volatile matter was affected by more rubberwood constituents being decomposed, especially hemicellulose to volatile matter. Additionally, temperature had a more significant effect on volatile matter content than residence time. For instance, by comparing the volatile matter content at 60 minutes of residence time at various torrefaction temperatures, it can be seen that the volatile matter content of the shredded rubberwood and the woodchip rubberwood more significantly decreased at 300 ºC compared to 260 ºC and 220 ºC with a reduction of 30% and 40%, respectively. This may be due to extensive devolatilization of cellulose and limited devolatilization of lignin at temperature higher than 250 °C, promoting faster and more extensive devolatilization [14]. The loss of moisture content from the rubberwood during

the torrefaction process is due to the release of both physical and bound moisture during drying. In this process, loss of hydroxyl groups in the rubberwood structure occurs and as a consequence, torrefied rubberwood becomes more hydrophobic, preventing absorption of moisture from the atmosphere [4,18].

3.4 Ultimate Analysis

Ultimate analysis was used to calculate the C, H, O, N, and S content of the torrefied shredded rubberwood and the woodchip rubberwood compared to raw rubberwood, lignite and subbituminous coal (Figure 6). In general, torrefaction leads to an increased carbon content of rubberwood while reducing oxygen and hydrogen contents [19,20].

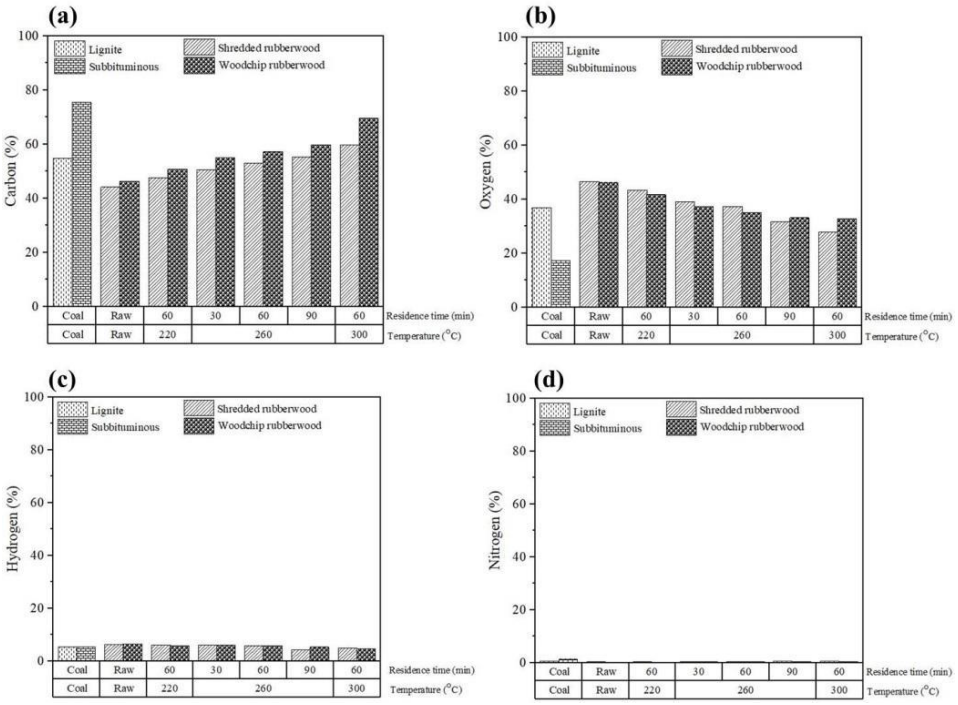

Figure 6 Ultimate analysis of the (a) carbon, (b) oxygen, (c) hydrogen, and (d) nitrogen contents of the torrefied shredded rubberwood and woodchip rubberwood compared to the raw shredded rubberwood and woodchip rubberwood, lignite, and subbituminous coal.

Based on Figure 6(a), the carbon content of the torrefied shredded rubberwood and the woodchip rubberwood increased by 9% and 12%, respectively. This is attributed to the release of volatile matter, including carbon, hydrogen, and oxygen. The released compounds contain a higher amount of hydrogen and

oxygen than carbon content, yielding an enhancement of the carbon content after torrefaction [21]. In addition, increasing the residence time period and torrefaction temperature promotes the devolatilization process, which resulted in a higher carbon content with an increase of 15% (Figure 6(a)), while the amount of oxygen and hydrogen content consistently decreased in the shredded rubberwood and woodchip rubberwood with a reduction of 23% (Figure 6(b)) and 15% (Figure 6(c)), respectively, compared to raw rubberwood. Since torrefaction was conducted under inert conditions, the changes of sulfur and nitrogen contents can be neglected (Figure 6(d)).

In order to classify the torrefied shredded (TS) and woodchip (TW) rubberwood, the atomic ratios of hydrogen to carbon (H/C) were plotted against oxygen to carbon (O/C), known as a Van Krevelen diagram, and compared to the raw shredded (RS) and woodchip (RW) rubberwood, lignite and subbituminous coal, as shown in Figure 7.

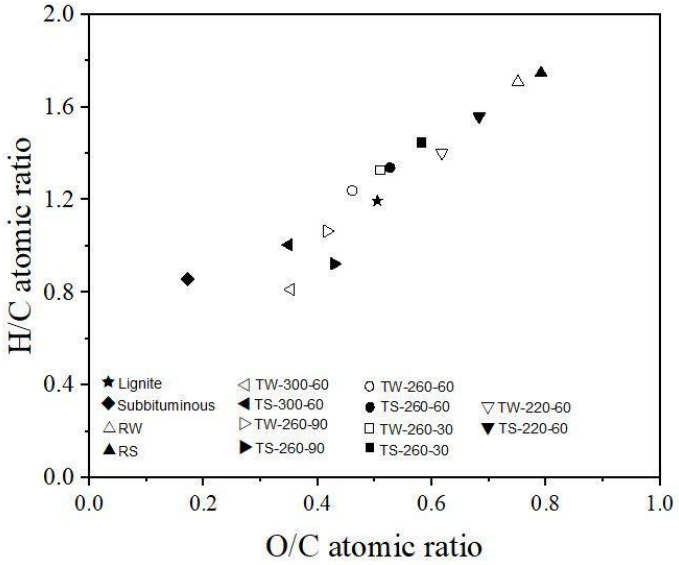

Figure 7 Van Krevelen plots of atomic H/C against O/C ratios for the torrefied shredded rubberwood (TS) and woodchip rubberwood (TW) compared to the raw shredded rubberwood (RS) and woodchip rubberwood (RW), lignite, and subbituminous coal.

The treatment with a low temperature and a short residence time of the shredded rubberwood and the woodchip rubberwood yielded high H/C and O/C ratios, close to those of raw rubberwood in the upper righthand corner of the Van Krevelen diagram. On the other hand, the treatment with a higher temperature and a longer residence time, moved the torrefied rubberwood close to lignite and

subbituminous coal in the lower left-hand corner of the Van Krevelen diagram. For instance, TW-300-60 and TS-300-60 have an O/C ratio of 0.353 and 0.348 and an H/C ratio of 0.81 and 1.81, respectively, which is in between the ranges of lignite and subbituminous with an O/C ratio of 0.504 and 0.172 and an H/C ratio of 1.195 and 0.857, respectively. During the torrefaction at a high temperature, rubberwood undergoes volatilization of hydrogen and oxygen atoms rather than carbon atoms, resulting in a lower H/C and O/C ratio [14].

3.5 HGI Analysis

One of the essential physical properties of coal, or biochar, for combustion/gasification is its grindability. This reflects its relative fracture, hardness and tenacity, which are influenced by coal rank, mineral matter and petrography. The Hardgrove Grindability Index (HGI) is a parameter of a material's grindability, which varies from 20 to 110, with a higher HGI value indicating that the material is easier to be grinded and less energy is required to reach the required degree of fineness. In this study, HGI analysis was done using the correlation of the elemental analysis result by following the empirical equation as evaluated by Chelgani, *et al.* [22,23]:

$$77.162 + 3.994\ ln\ \ln(S) - 10.92(H) + 1.904(Moisture) - 0.424(Ash) - 11.765ln(\frac{O+N}{C}) \quad (1)$$

Figure 8 shows the HGI results for biochar of the torrefied shredded rubberwood and woodchip rubberwood. The raw lignite coal, subbituminous coal, shredded, and woodchip rubberwood had HGI values of 53, 69, 43, and 44, respectively. It is interesting to note that the torrefied woodchip rubberwood treated at a temperature of 300 °C with a residence time of 60 minutes, which had the highest GCV and the lowest volatile matter among the tested rubberwood samples, had the highest HGI (47). This is due to the fact that an increase of the torrefaction temperature results in more degradation of hemicellulose, thus the loss of fibrous structure and tenacity of the biomass is higher, making the grinding easier [3,24]. During the torrefaction process, hemicellulose experiences significant decomposition. As the hemicellulose content begins to decrease, with increasing torrefaction temperature the grinding characteristics improve and the HGI value increases. Moreover, the residence time spent in the torrefier is another important parameter that affects the grindability of the material. It should be noted that longer residence times at a torrefaction temperature of 260 °C improved the grindability of the shredded and woodchip rubberwood by 13% and 7%, respectively, which is not to the extent that was observed for torrefaction temperature (approximately 20%). Based on Figure 8 it can be concluded that a clear relationship exists between torrefaction temperature, residence time and

HGI. Increasing the torrefaction temperature and residence time concomitantly increases the HGI value, indicating low resistance to grinding.

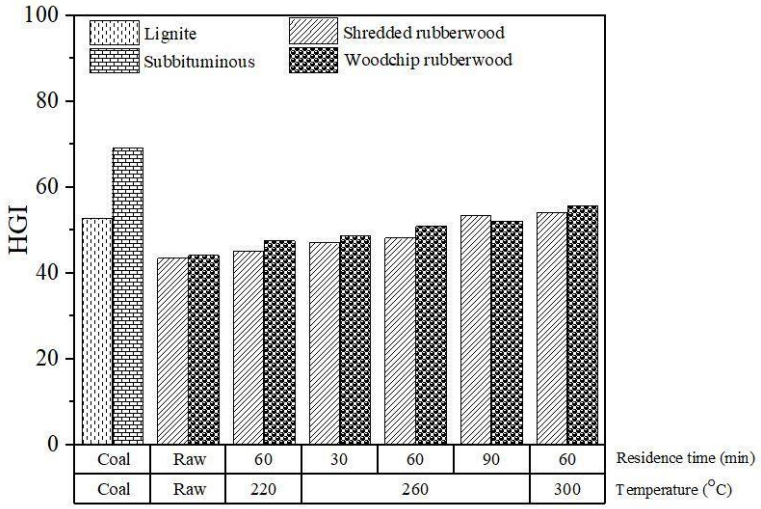

Figure 8 Calculated HGI values of the torrefied shredded rubberwood and woodchip rubberwood compared to the raw shredded rubberwood and woodchip rubberwood, lignite, and subbituminous coal.

4 Conclusion

Two different forms of rubberwood waste, i.e. shredded rubberwood and woodchip rubberwood, were torrefied in a vertical tubular reactor. The results demonstrated that the torrefaction process was affected by particle size, temperature and residence time.

The biochar resulted from the torrefaction treatment of the woodchip rubberwood with a temperature of 300 °C and a residence time of 60 min had a calorific value and HGI similar to those of subbituminous coal, i.e. a GCV value of 6313 kcal/kg and an HGI value of 47. This study showed the advantage of using torrefaction in eliciting biomass characteristics and also provides design guidelines for fundamental prospects for boosting the coal rank by mixing biomass and coal.

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